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FREQUENCY MODULATED NMR SPECTROMETER
FOR MEASUREMENT OF INTERNAL MAGNETIC FIELDS

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MENT OF INTERNAL MAGNETIC FIELDS

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Introduction

For the investigation of nuclear magnetic resonances in ordered magnetic materials with zero applied magnetic field it is necessary to modulate and sweep the frequency of the nuclear magnetic resonance absorption circuit. The frequency modulation yields spurious amplitude modulation, and wide frequency sweeps under these circumstances would normally result in a base line shift at the output of the tuned amplifier stage. Many spectrometers are described in the literature for the measurements of hyperfine fields in magnetic materials or for the detection quadrupole resonances /Kushida 1956, Verdick 1961, Vilson 1964/. To reduce the effects of spurious amplitude modulation the amplifier and phase detector are usually tuned to the second harmonic of the modulation frequency. .

In the NMR spectrometer described here, a Robinson-type oscillator /Robinson 1959/, chosen for its simple construction and sensitivity is used. Because of the high operating frequencies /25-100 MHz/ the first amplifier stage is a tuned anode type circuit. By synchronously tuning the grid and anode capacitor the anode inductor automatically tunes to the exact grid resonant frequency and the level of oscillation is kept constant even during a wide sweep.

The NMR signal of ^{57}Fe detected in first harmonic in iron and iron-cobalt alloys demonstrates the performance of the equipment.

The circuit

The block diagram of the spectrometer and the circuit diagram of the oscillator are shown in Figs 1 and 2. Both the tuned amplifier and the limiter are double triodes in cascade circuit. Owing to the lower input capacitance and higher input electronic resistance, the cascade circuit is preferable at higher frequencies to the usual pentode in the Robinson circuit. It is not susceptible

to self-oscillation and the nearly equal capacities of grid and anode achieve a synchronous run of the two tuning circuits even for large sweep amplitudes. The frequency is tuned by a motor-driven rotary capacitor, or, over a small frequency interval, by a slow sweep applied to the varicap. The frequency is modulated by means of a second varicap. If the resonant frequency of the anode circuit is appropriately chosen, for the absorption circuit the amplitude modulation signal at the demodulator output will be minimized /Wilson 1964/ so as to keep the oscillation level constant during the tuning, provided the minimum condition is automatically reset by the AFC circuit. By means of a second selective amplifier and phase detector tuned to the modulation frequency, the bias to the premagnetizing coil of the ferrite cored inductor of the anode circuit is always so adjusted as to keep the amplitude modulation at a minimum during tuning. The P.I.D. control included in the automatic frequency control loop ensures a fast and smooth A.F.C. control characteristic. If the frequencies of the two tuned circuit do not deviate too far from each other, the inductance of the anode is automatically adjusted by the A.F.C. circuit to the desired frequency. To facilitate the synchronization, the d.c. circuit of the A.F.C. can be switched off and the input bias to the premagnetizing amplifier can be adjusted by the potentiometer for the maximum oscillation level while the two tuned circuits are being synchronized. The frequency of the anode resonance circuit for the minimum harmonic component will be then somewhat higher /Wilson 1964/ but it will be automatically set if the A.F.C. circuit is switched on.

The signal-to-noise ratio is somewhat better with the d.c. output signal taken from the Ge diode in the anode circuit than that which is usually taken from the grid circuit of the limiter.

The rf-amplitude is varied by the D.C. bias of the limiter anode circuit.

Performance

We have used the absorption circuit to study the hyperfine field of ^{57}Fe in iron and iron-based alloys. To illustrate the sensitivity and the use of the equipment, the NMR signal of ^{57}Fe detected at first harmonic in 0,5 g of well annealed natural iron powder of 5N purity /grain size of particles smaller than 56μ / is shown in Fig.3. The modulation frequency is 70 c/s, the modulation amplitude 5 kc/s. The lock-in time constant for the A.F.C. circuit is 100 sec, that for

the detector 30 sec, and the time constants of D and I in the P.I.D. control are 0,03 and 3 sec, respectively. The line shape is characteristic of a mixed absorption and dispersion component. The enhancement factor can be determined by measuring the line shape at different amplitudes. /Streever 1963./ Using the relaxation times as measured by spin-echo measurements /Cowan 1964/, we get $\eta = 7000$.

At higher modulation frequencies and 90° detection with respect to the modulation signal an excellent signal-to-noise ratio and a flat base line over large frequency range can be obtained. Though all the details of the adiabatic fast passage /Cowan 1964/ couldn't be followed, and the line width of iron was slightly dependent on the modulation amplitude, the improved signal-to-noise ratio makes it possible to detect weak satellites beside the central resonance due to certain neighbours of the impurities in the iron matrix. Fig. 4. shows the hyperfine field distribution measured in Fe-0,72 at% Co alloy at room temperature. The satellites observed on the high frequency side of the central resonance correspond to the second, third and fourth neighbours of the Co impurities /Grüner 1971/.

The sample coil is kept in a probe at temperatures variable by gas stream temperature controller /Balla 1968/, and has been used to measure the temperature dependence of the hyperfine field in the $-190...+150^\circ\text{C}$ range in iron and iron-based alloys.

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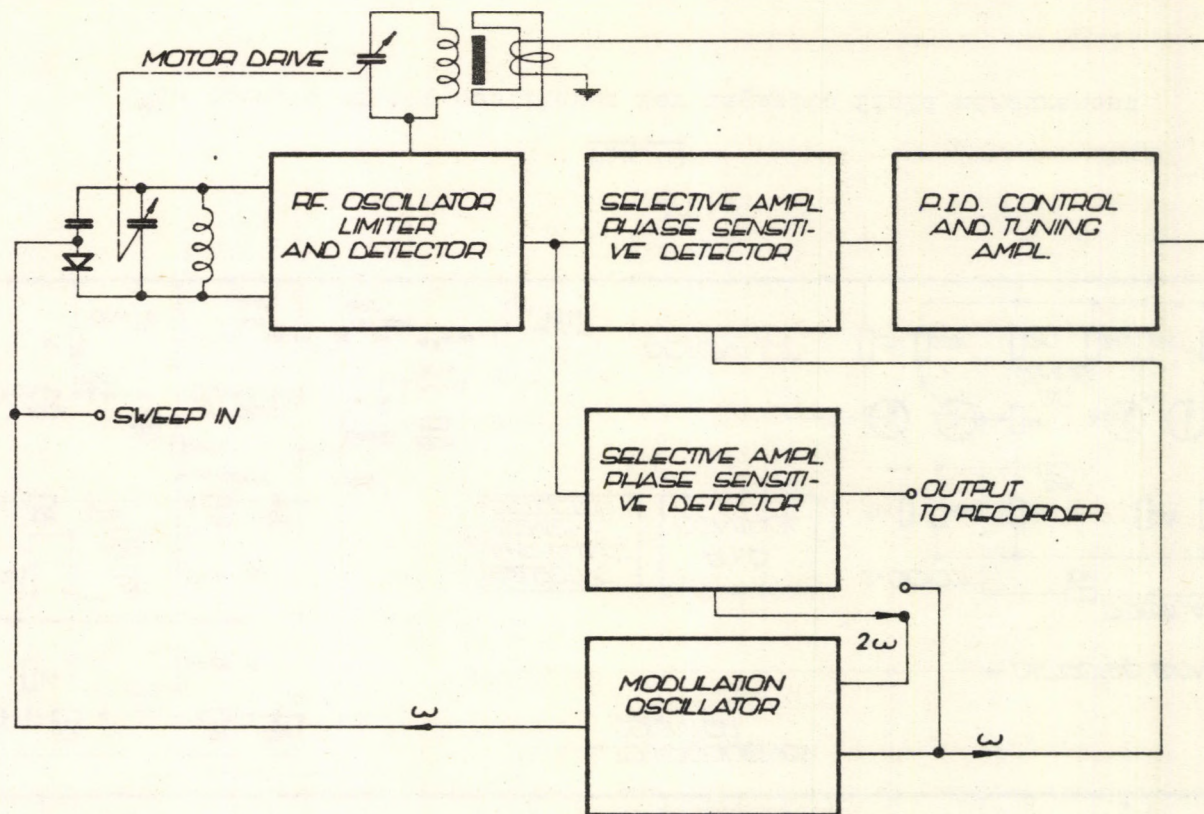


Fig. 1

NMR Spectrometer for the measurement of internal magnetic fields
with operating frequencies from 25 MHz to 100 MHz

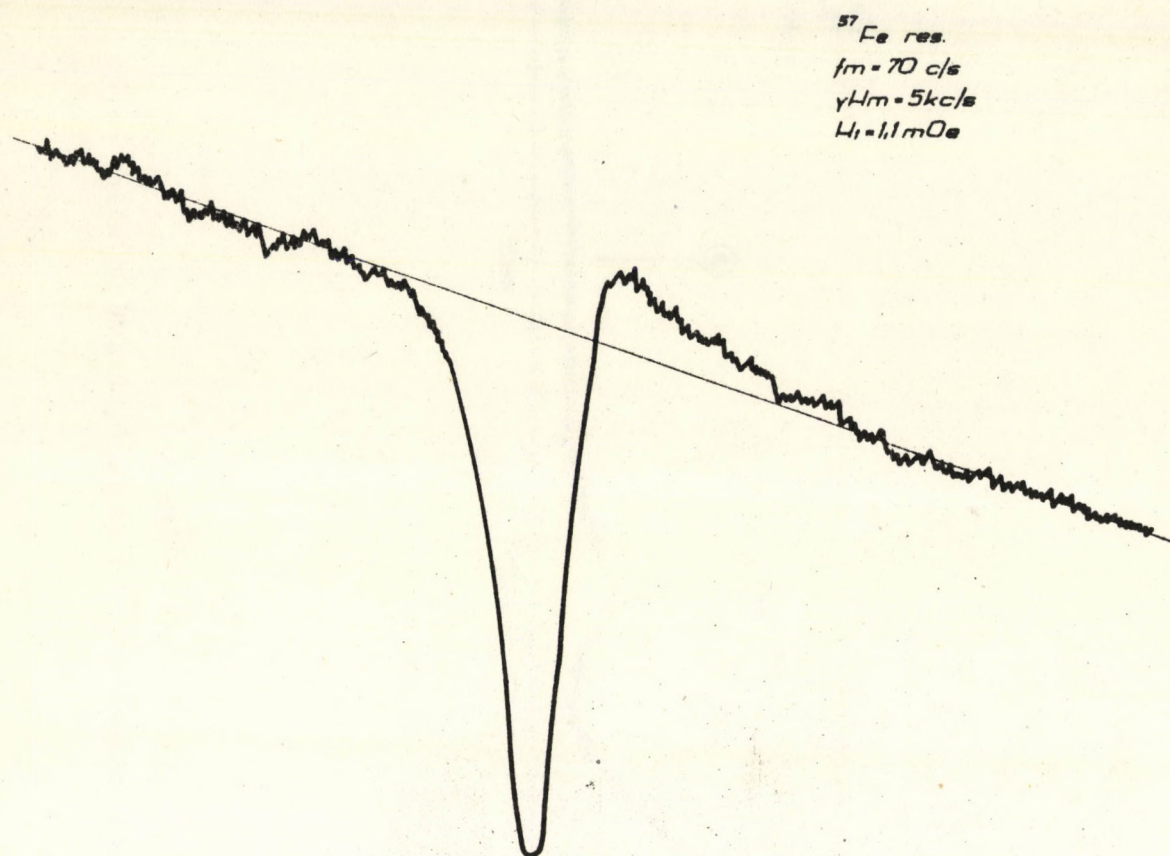


Fig. 3

NMR signal of ^{57}Fe in natural iron at low rf amplitude

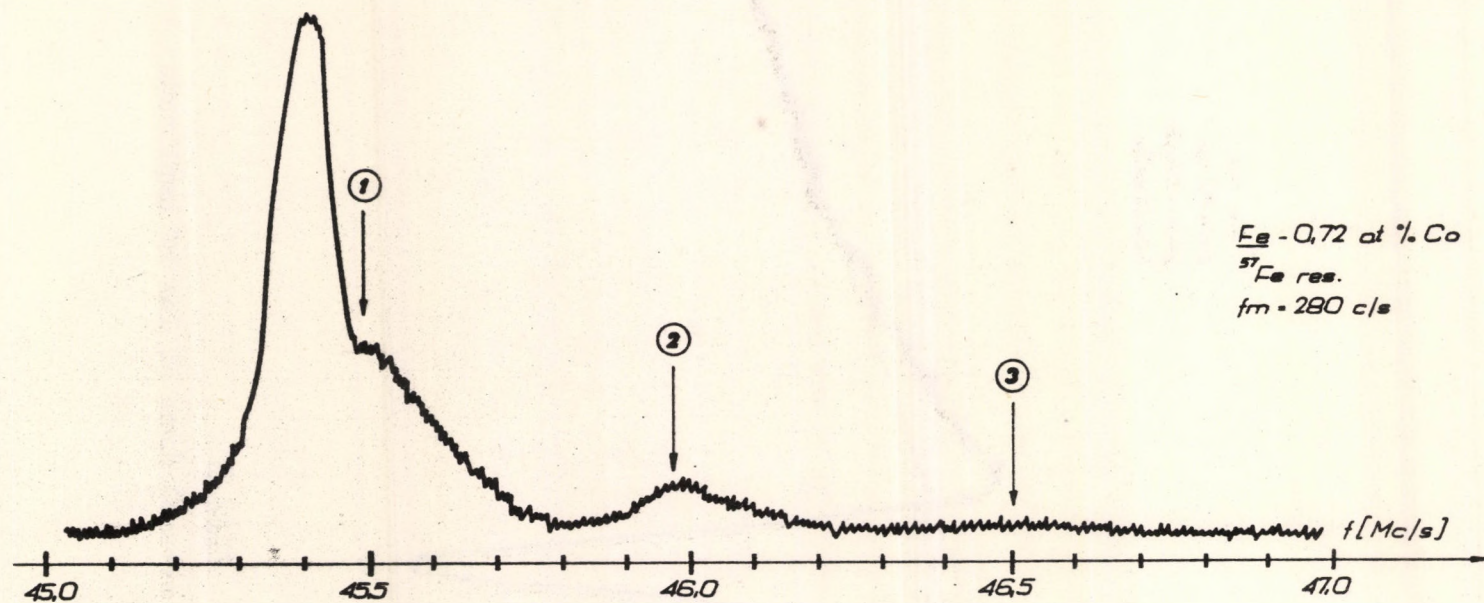


Fig. 4
NMR signal of ^{57}Fe in Fe-0,72 at% Co alloy

ABSTRACT

A Robinson-type oscillator with an automatic frequency control circuit, suitable for the measurement of internal magnetic fields, is described. An AFC circuit ensures that the tuning of the tank circuit is such that the first harmonic of the modulation frequency is kept at a minimum in the detector output while the NMR signal is detected at the first harmonic. Cascade amplifier stages permit operation at higher frequencies with a better signal-to-noise ratio and larger sweep amplitudes than with other types of amplifiers.

The use of the equipment is demonstrated by measuring the ^{57}Fe NMR signal in iron and iron-cobalt alloys.

РЕЗЮМЕ

В работе описывается осциллятор типа Робинсона с автоматической схемой управления частоты, разработанный для измерения внутренних магнитных полей. Частотная модуляция сопровождается амплитудной модуляцией, низкий уровень этого эффекта обеспечивается схемой AFC, причем сигнал ЯМР измеряется на первой гармонике. Ступени каскадного усилителя позволяют действие осциллятора и на более высоких частотах с лучшим отношением сигнала к шуму, а также с большими амплитудами развертки, чем другие типы осцилляторов. В качестве примера действия осциллятора описывается измерение сигнала ЯМР ядер ^{57}Fe в железе и сплавах железа-кобальта.

KIVONAT

A cikk belső mágneses tér mérésére alkalmas automatikus frekvenciaszabályozású Robinson-oszcillátort ismertet. A frekvenciamoduláció következtében fellépő amplitúdómodulációs szintet AFC áramkör tartja alacsony szinten, ezért az NMR jeltartalom első harmonikusa detektálható. A kaszkád erősítőfokozatok következtében az oszcillátor átfogható frekvenciatartománya nagy, jel/zaj viszonya jobb, a frekvenciamoduláció mértéke nagyobb, mint a szokásos megoldásoknál.

A berendezés tulajdonságait vas és vas-kobalt ötvözetben detektált ^{57}Fe NMR jel demonstrálja.

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